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⑭ 光ファイバ母材の製造方法

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明 細 書

発明の名称 光ファイバ母材の製造方法

特許請求の範囲

1. 超音波振動を利用してガラス組成物液体原料を霧化させ、この霧化粒子を酸素および水を含むガスと共に混合、燃焼してガラス燐を含んだ火炎を発生させてターゲット上に衝突させガラスブロックを堆積させることを特徴とする光ファイバ母材の製造方法。

発明の詳細な説明

先に、本発明者はガラス組成物液体原料を水を含有するガスで霧状にした後、酸化性ガスと混合して燃焼させ、この火炎をターゲット上に吹付けることにより光ファイバ用ガラスブロックを堆積させる方法を提案した。この方法はガラスブロックを高速度、かつ高収率で堆積でき、しかも連続的に製造することができるという特徴をもっている。しかし、ガラスブロックを100g/hr以上の超高速度で堆積させようとするとガス圧が高くなるために霧化粒子の速度が遅くなり、ターゲット

への堆積収率が低下してくるという問題点がある。

本発明は上記問題点を解決させるために、ガラス組成物液体原料を超音波振動のような電気機械トランスジューサを利用して霧化させる方法を提供するものである。すなわち、超音波振動子に振動拡大ホーンを取付け、この振動拡大ホーンの前端面に開口するガラス組成物液体原料供給孔に上記液体を供給する装置を設けて超音波振動により振動拡大ホーンの前端面に到達した上記液体を霧化させ、この霧化粒子を酸素および水を含有するガスと共に混合、燃焼してガラス燐を含んだ火炎を発生させる。そしてこの火炎をターゲット上に衝突させてガラスブロックを堆積させる方法である。

以下図面を用いて本発明の方法を説明する。

第1図は本発明のガラスブロック製造方法を説明するための概略図である。1は超音波振動子で、その上端に振動拡大ホーン2を固定している。超音波振動子1および振動拡大ホーン2はともに、振動拡大ホーン2の振動が零なる位置、すなわち振動の節部14に固定した複数の支柱15を介し

(2)

(1)

て内側ガス供給管7内に配置されている。この内側ガス供給管7内には矢印10方向からH₂、あるいはN₂、He、Ar、Neなどのうち少なくとも1種以上のガスを含んだH₂ガスが送り込まれ、ノズル7'から噴射されるようになっている。また内側ガス供給管7は不活性ガス供給管8と酸化性ガス供給管9で覆われ、これらの供給管は同心状の3重管構造になっている。不活性ガス供給管8内には矢印11方向からN₂、Ar、He、Neなどの不活性ガスが送り込まれ、ノズル8'から噴射されるようになっている。酸化性ガス供給管9内には矢印12方向からO₂、CO₂、NO₂、空気、オゾンなどの酸化性ガス、あるいはSiC₄、BF₃などのガラス原料の蒸気を含んだ上記酸化性ガスが送り込まれ、ノズル9'から噴射されるようになっている。ノズル7'、8'、9'は同心状の3重管構造になっている。4は超音波発振器3と超音波振動子1とを結ぶ導線、5はガラス組成物液体原料供給装置で、供給管6を介して、振動拡大ホーン2の前端面に開口

(5)



ラス化したロッド17を増積させるようにしたものである。

第2図は、超音波振動を利用して液体原料を酸化させる方法と、先に本発明者が提案した水素を含むガスで液体原料を酸化させる方法を併用したことを特徴とするガラスブロッタの製造方法を説明するための概略図である。この方法は液体の酸化量を超音波振動子に加える電気的エネルギー量と水素を含むガス流量の両方で制御することができるのでより自由度が増えるという特徴を有している。すなわち、水素を含むガスを供給装置21からガス供給管22を介して振動拡大ホーン2の前端面に開口するガス供給孔13'へ送り込むことによつてガラス組成物液体原料供給装置5からの液体原料を供給管6を介して強制的にガラス組成物液体原料供給孔13'へ吸い上げさせて酸化させると共に、超音波振動の加振によつても液体を酸化させるものである。その結果、第1図の場合よりも液体の酸化量を増大させることができる。なお、矢印10'方向からは不活性ガスを、矢印

(5)

するガラス組成物液体原料供給孔13と連結している。ガラス組成物液体原料供給装置5から供給管6を経て振動拡大ホーン2の前端面13に到達した液体原料は表面張力によつて薄い液膜を形成するが、超音波振動の加振の影響を受けて微細粒子となり、前方へ飛散する。この場合本作用による微細粒子の性質は、超音波振動子により電気的エネルギーを振動エネルギーに変換し、この振動を利用して液体原料を酸化させるものであるために、粒子速度ならびに運動エネルギーが非常に小さい。したがつて前方へ飛散した微細粒子は矢印10方向から供給されたH₂ガスで搬送され、ノズル7'より噴射される。そしてノズル出口部でこのH₂ガスに点火して燃焼させ、ついで矢印11方向および矢印12方向からそれぞれ、不活性ガスおよび酸化性ガスを流すことにより、3重管ノズルの前方にガラス膜を含んだ火炎16を生じさせる。この火炎を矢印20方向に回転しつつ矢印19方向に移動するターゲット18に吹付けることによりターゲット上にガラス膜あるいはガ

(4)

11'方向からは酸化性ガスを流す。本発明に適用できるガラス組成物液体原料は、アルキル化合物、ハロゲン化合物、水素化合物からなるシリコン化合物、および屈折率制御用化合物を含んだ上記シリコン化合物、アルコールとか水に溶解あるいは分散したシリコン化合物、などである。本発明に用いる超音波振動子としては、実施例で示したように、磁歪形振動子、あるいは電歪形振動子を用いることができる。振動子の周波数、入力電力は液体の酸化量によつて決めることができる。振動子の周波数は通常、数KH.~100KH.に設定する。入力電力は10数W~数百Wを用いる。また液体の酸化量は、液体の表面張力値、拡大ホーンの液膜の周縁に接触するホーン材質、液体とその材質との親和性、ホーンの構造などによつてもちがつてくる。ホーンの構造としては、エキスポネンシヤル型、コニカル型、単純段付ホーン、さらにはこれらを組み合わせた複合タイプを用いることができる。

第3図は第2図の装置において、液体にテトラ

(6)

エトキシシラン81 (OC_2H_5)₂を用い、拡大ホーンをアルミニウムで形成した場合の超音波振動子への入力電力と液体の霧化量との関係の一例を示したものである。これはエキスポネンシャル型のホーンを用い、20 KHzの周波数で振動させた結果である。ただし、21から送り込む H_2 ガス流量2 L/min、矢印10'から送り込む Ar ガス流量2.5 L/min、矢印11'から送り込む O_2 ガス流量5 L/min、である。同図からわかるように、入力電力を大きくすることによつて液体の霧化量を増やすことができることを示している。第4図はターゲットとして底が半球球を有する円筒管(外径70 mm ϕ)にガラス煤を堆積させた結果である。これは第3図の結果に対応したものである。入力電力10数Wで100 g/hrのガラス煤堆積速度を示しており、入力電力20数Wで400 g/hr程度の超高速ガラス煤堆積速度を得ることができた。従来方法でこのような超音速堆積速度を得ようとする、堆積収率が10%以下になるのに対し、本発明の方法では20%程度の堆積収率

(7)

が実現されている。

以上述べたごとく、本発明の方法によると、電気的エネルギー量で液体の霧化量を制御することができ、かつ制御量も極めて広範囲に変えられ、さらに従来法のように霧化量を多くするためにガス圧を高くしなくてもよい。したがって、霧化粒子の速度が速くならないためにターゲットへの堆積収率を上げることができる。

図面の簡単な説明

第1図および第2図は本発明の実施例で用いる光ファイバ母材製造装置の概略断面図、第3図は超音波振動子への入力電力と液体の霧化量との関係を示すグラフ、第4図は超音波振動子への入力電力とガラス煤の堆積速度との関係を示すグラフである。

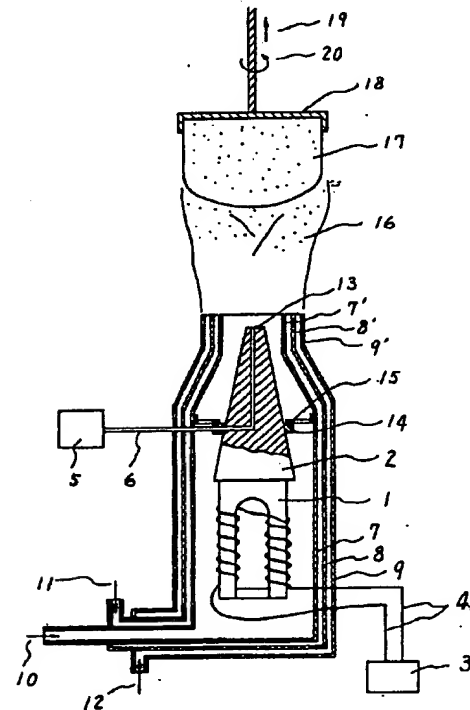
各図において、1は超音波振動子、2は振動拡大ホーン、5はガラス組成物液体原料供給装置、10は H_2 を含むガスの導入方向、11は不活性ガスの導入方向、12は酸化性ガスの導入方向、13はガラス組成物液体原料供給孔、17はガラ

(8)

スロッド、18はターゲット、21は H_2 を含むガスの供給装置である。

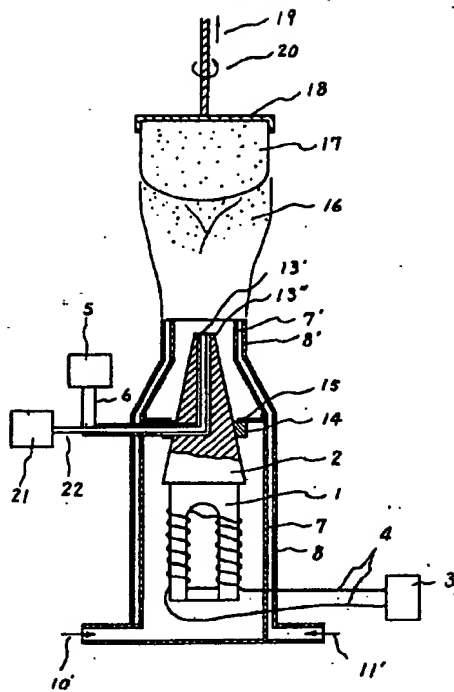
代理人 弁理士 薄田利幸

第1図

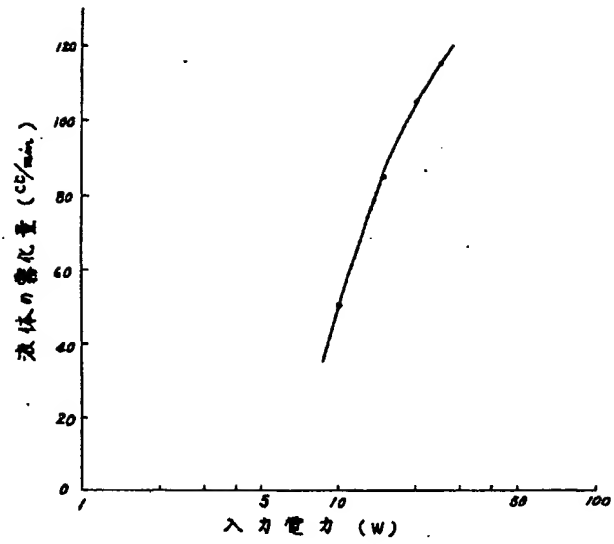


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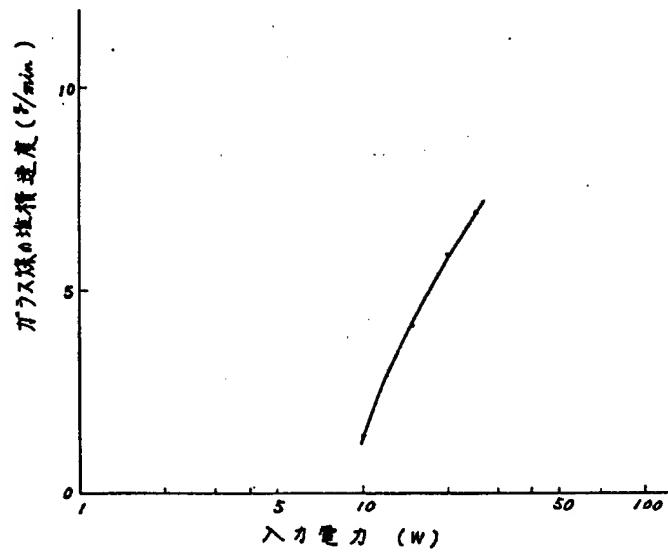
第 2 図



第 3 図



第 4 図



PATENT COOPERATION TREATY

PCT

INTERNATIONAL PRELIMINARY EXAMINATION REPORT

(PCT Article 36 and Rule 70)

14
REC'D 17 NOV 2000

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PCT

Applicant's or agent's file reference HAWTOF 7-2	FOR FURTHER ACTION See Notification of Transmittal of International Preliminary Examination Report (Form PCT/IPEA/416)	
International application No. PCT/US99/16616	International filing date (day/month/year) 22 JULY 1999	Priority date (day/month/year) 07 AUGUST 1998
International Patent Classification (IPC) or national classification and IPC IPC(7): CO3B 20/00 and US Cl.: 65/17.4		
Applicant CORNING INCORPORATED		

1. This international preliminary examination report has been prepared by this International Preliminary Examining Authority and is transmitted to the applicant according to Article 36.
2. This REPORT consists of a total of 4 sheets.
- ☐ This report is also accompanied by ANNEXES, i.e., sheets of the description, claims and/or drawings which have been amended and are the basis for this report and/or sheets containing rectifications made before this Authority. (see Rule 70.16 and Section 607 of the Administrative Instructions under the PCT).

These annexes consist of a total of 0 sheets.

3. This report contains indications relating to the following items:

- I ☒ Basis of the report
- II ☐ Priority
- III ☐ Non-establishment of report with regard to novelty, inventive step or industrial applicability
- IV ☐ Lack of unity of invention
- V ☒ Reasoned statement under Article 35(2) with regard to novelty, inventive step or industrial applicability; citations and explanations supporting such statement
- VI ☐ Certain documents cited
- VII ☐ Certain defects in the international application
- VIII ☐ Certain observations on the international application

Date of submission of the demand 04 FEBRUARY 2000	Date of completion of this report 23 OCTOBER 2000
Name and mailing address of the IPEA/US Commissioner of Patents and Trademarks Box PCT Washington, D.C. 20231	Authorized officer SEAN VINCENT DEBORAH THOMAS PARALEGAL SPECIALIST
Facsimile No. (703) 305-3230	Telephone No. (703) 308-0661

INTERNATIONAL PRELIMINARY EXAMINATION REPORT

International application No.

PCT/US99/16616

I. Basis of the report

1. With regard to the elements of the international application:*

☒ the international application as originally filed☒ the description:

pages 1-15, as originally filed

pages NONE, filed with the demand

pages NONE, filed with the letter of

☒ the claims:

pages 16-18, as originally filed

pages NONE, as amended (together with any statement) under Article 19

pages NONE, filed with the demand

pages NONE, filed with the letter of

☒ the drawings:

pages 1-7, as originally filed

pages NONE, filed with the demand

pages NONE, filed with the letter of

☒ the sequence listing part of the description:

pages NONE, as originally filed

pages NONE, filed with the demand

pages NONE, filed with the letter of

2. With regard to the language, all the elements marked above were available or furnished to this Authority in the language in which the international application was filed, unless otherwise indicated under this item.
These elements were available or furnished to this Authority in the following language _____ which is:☐ the language of a translation furnished for the purposes of international search (under Rule 23.1(b)).☐ the language of publication of the international application (under Rule 48.3(b)).☐ the language of the translation furnished for the purposes of international preliminary examination (under Rules 55.2 and/ or 55.3).

3. With regard to any nucleotide and/or amino acid sequence disclosed in the international application, the international preliminary examination was carried out on the basis of the sequence listing:

☐ contained in the international application in printed form.☐ filed together with the international application in computer readable form.☐ furnished subsequently to this Authority in written form.☐ furnished subsequently to this Authority in computer readable form.☐ The statement that the subsequently furnished written sequence listing does not go beyond the disclosure in the international application as filed has been furnished.☐ The statement that the information recorded in computer readable form is identical to the written sequence listing has been furnished.4. ☒ The amendments have resulted in the cancellation of:☒ the description, pages NONE☒ the claims, Nos. NONE☒ the drawings, sheets/fig. NONE5. ☒ This report has been drawn as if (some of) the amendments had not been made, since they have been considered to go beyond the disclosure as filed, as indicated in the Supplemental Box (Rule 70.2(c)).**

* Replacement sheets which have been furnished to the receiving Office in response to an invitation under Article 14 are referred to in this report as "originally filed" and are not annexed to this report since they do not contain amendments (Rules 70.16 and 70.17).

**Any replacement sheet containing such amendments must be referred to under item 1 and annexed to this report.

INTERNATIONAL PRELIMINARY EXAMINATION REPORT

International application No.

PCT/US99/16616

V. Reasoned statement under Article 35(2) with regard to novelty, inventive step or industrial applicability; citations and explanations supporting such statement**1. statement**

Novelty (N)	Claims	<u>2, 13-16 and 18-21</u>	YES
	Claims	<u>1, 3-12 and 17</u>	NO
Inventive Step (IS)	Claims	<u>none</u>	YES
	Claims	<u>1-21</u>	NO
Industrial Applicability (IA)	Claims	<u>1-21</u>	YES
	Claims	<u>none</u>	NO

2. citations and explanations (Rule 70.7)

Claims 1, 3-12 and 17 lack novelty under PCT Article 33(2) as being anticipated by Hitachi.

Hitachi teaches a burner and a method of forming silica soot by delivering liquid siloxane ($\text{Si}(\text{OC}_2\text{H}_5)_4$) precursor to an injector recessed within a burner, discharging liquid precursor through a liquid orifice insert into a frustoconical chamber in the burner recess, introducing inert gases including nitrogen and oxygen into the chamber, discharging the precursor from the burner atomization orifice as an aerosol and reacting the aerosol in a burner flame (see English abstract as well as the figures and sections 3 and 7 of the Japanese text).

Claims 2, 13-16 and 18-21 lack an inventive step under PCT Article 33(3) as being obvious over Hitachi.

Hitachi does not teach a precision orifice less than 0.011 inches in diameter. It would have been obvious to make the precision orifice of Hitachi 0.011 inches in diameter because it would have been an optimal size for the particular conditions at the time.

Hitachi does not teach releasable engagement between the liquid orifice insert and the liquid tube. It would have been obvious to make the tube releasable from the insert because it would have aided in the replacement of inserts.

Hitachi does not teach a plurality of atomization orifices circumferentially spaced around the liquid orifice insert. It would have been obvious to include a plurality of atomization orifices as such because it would appear to be no more than a duplication of the exact same orifice with no new results.

Hitachi does not teach that the insert was a jewel or a precision orifice material. It would have been obvious to use a jewel or a precision orifice material because the ultrasonic atomization means of Hitachi would have necessarily included the use of special materials capable of withstanding intense vibrations.

(Continued on Supplemental Sheet.)

INTERNATIONAL PRELIMINARY EXAMINATION REPORT

International application No.

PCT/US99/16616

Supplemental Box

(To be used when the space in any of the preceding boxes is not sufficient)

Continuation of: Boxes I - VIII

Sheet 10

I. BASIS OF REPORT:

5. (Some) amendments are considered to go beyond the disclosure as filed:
NONE

V. 2. REASONED STATEMENTS - CITATIONS AND EXPLANATIONS (Continued):

Hitachi does not specifically teach shaping the atomization orifice to reduce turbulence. It would have been obvious to shape the atomization orifice of Hitachi to reduce turbulence because complete combustion would have been hampered by turbulent flow of combustion gases back into the burner recess.

Hitachi does not teach a rounded orifice rim or a particular radius of round. It would have been obvious to make the rim of the atomization orifice rounded because it would have been a mere matter of design choice. The claimed radius limitations would not have been critical but merely optimal for the particular conditions at the time.

Claims 1-21 meet the criteria set out in PCT Article 33(4), because the method of forming soot and the burner would have been useful in industry.

----- NEW CITATIONS -----
NONE

PCTWORLD INTELLECTUAL PROPERTY ORGANIZATION
International Bureau

INTERNATIONAL APPLICATION PUBLISHED UNDER THE PATENT COOPERATION TREATY (PCT)

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		(43) International Publication Date: 17 February 2000 (17.02.00)

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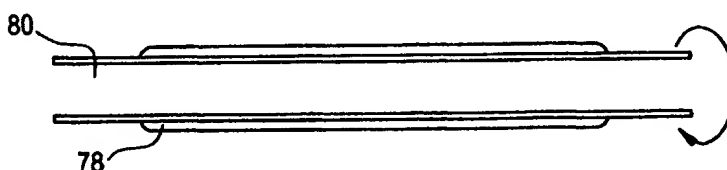
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(81) Designated States: AL, AM, AT, AU, AZ, BA, BB, BG, BR, BY, CA, CH, CN, CU, CZ, DE, DK, EE, ES, FI, GB, GE, GH, GM, HR, HU, ID, IL, IS, JP, KE, KG, KP, LS, LT, LU, LV, MD, MG, MK, MN, MW, MX, NO, NZ, PL, PT, RO, RU, SD, SE, SG, SI, SK, SL, TJ, TM, TR, TT, UA, UG, US, UZ, VN, YU, ZW, European patent (AT, BE, CH, CY, DE, DK, ES, FI, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE).

Published*With international search report.*

(54) Title: METHOD AND APPARATUS FOR FORMING SOOT FOR THE MANUFACTURE OF GLASS

**(57) Abstract**

The present invention is directed to a method and apparatus for forming soot used in making glass, and in particular, optical waveguides. A liquid precursor (66) is first fed into orifice (52) of a liquid orifice insert (48) within an injector (44) positioned within an atomizing burner assembly, and is thereafter discharged from the injector into a pressurization chamber (56). An atomization gas (70) is also fed into the pressurization chamber (56) to mix with the liquid precursor liquid stream (68) which breaks into droplets (76). The liquid precursor and atomization gas are forced under pressure out of an atomization orifice (32) on the face of the burner (30) assembly. Flame gas (74), reaction gas (84) and shield gas (82) are ejected from burner orifices (40, 38, 36 and 34) to produce the flame. The atomized liquid precursor thus discharged is fed into the flame (72) produced at the face of the burner assembly where the atomized liquid precursor reacts with the flame to form soot (78) on a rotating mandrel (80).

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BY	Belarus	IS	Iceland	MX	Mexico	US	United States of America
CA	Canada	IT	Italy	NE	Niger	UZ	Uzbekistan
CF	Central African Republic	JP	Japan	NL	Netherlands	VN	Viet Nam
CG	Congo	KE	Kenya	NO	Norway	YU	Yugoslavia
CH	Switzerland	KG	Kyrgyzstan	NZ	New Zealand	ZW	Zimbabwe
CI	Côte d'Ivoire	KP	Democratic People's Republic of Korea	PL	Poland		
CM	Cameroon	KR	Republic of Korea	PT	Portugal		
CN	China	KZ	Kazakistan	RO	Romania		
CU	Cuba	LC	Saint Lucia	RU	Russian Federation		
CZ	Czech Republic	LI	Liechtenstein	SD	Sudan		
DE	Germany	LK	Sri Lanka	SE	Sweden		
DK	Denmark	LR	Liberia	SG	Singapore		
EE	Estonia						

INTERNATIONAL SEARCH REPORT

International application No.
PCT/US99/16616

A. CLASSIFICATION OF SUBJECT MATTER

IPC(6) :CO3B 20/00

US CL :65/17.4

According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)

U.S. : 65/17.2, 17.4, 414, 421; 423/336, 337

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practicable, search terms used)

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X	US 5,622,750 A (KILIAN ET AL) 22 April 1997, figures 1-3, col. 3, lines 22-65, col. 5, lines 7-62, col. 6, lines 8-42.	1, 3, 5, 7, 10 and 19
----		----
Y		2, 4, 6, 8, 9, 18, 20 and 21
X	JP 55-23067 A (NIPPON TELEGR & TELEPH CORP) 19 February 1980, abstract, figure.	11
----		----
Y		12-18
X	JP 56-14438 A (HITACHI LTD) 12 February 1981, abstract, figures, p. 3.	1-8, 10-12 and 17
----		----
Y		9, 13-16 and 18-21

☒ Further documents are listed in the continuation of Box C. ☐ See patent family annex.

* Special categories of cited documents:	*T* later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention
A document defining the general state of the art which is not considered to be of particular relevance	*X* document of particular relevance; the claimed invention cannot be considered novel or cannot be considered to involve an inventive step when the document is taken alone
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L document which may throw doubts on priority claim(s) or which is cited to establish the publication date of another citation or other special reason (as specified)	*A* document member of the same patent family
O document referring to an oral disclosure, use, exhibition or other means	
P document published prior to the international filing date but later than the priority date claimed	

Date of the actual completion of the international search

19 SEPTEMBER 1999

Date of mailing of the international search report

04 OCT 1999

Name and mailing address of the ISA/US
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Washington, D.C. 20231
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INTERNATIONAL SEARCH REPORT

International application No.
PCT/US99/16616

C (Continuation). DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
Y	US 5,043,002 A (DOBBINS ET AL) 27 August 1991, col. 5, lines 60-65.	9
A	US 5,788,730 A (RUPPERT ET AL) 04 August 1998.	1-21
A	US 5,110,335 A (MILLER ET AL) 05 May 1992.	1-21
A	US 5,108,665 A (CROOKER ET AL) 28 April 1992.	1-21

INTERNATIONAL SEARCH REPORT

International application No.

PCT/US99/16616

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IPC(6) :CO3B 20/00

US CL :65/17.4

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Electronic data base consulted during the international search (name of data base and, where practicable, search terms used)

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X ----- Y	US 5,622,750 A (KILIAN ET AL) 22 April 1997, figures 1-3, col. 3, lines 22-65, col. 5, lines 7-62, col. 6, lines 8-42.	1, 3, 5, 7, 10 and 19 ----- 2, 4, 6, 8, 9, 18, 20 and 21
X ----- Y	JP 55-23067 A (NIPPON TELEGR & TELEPH CORP) 19 February 1980, abstract, figure.	11 ----- 12-18
X ----- Y	JP 56-14438 A (HITACHI LTD) 12 February 1981, abstract, figures, p. 3.	1-8, 10-12 and 17 ----- 9, 13-16 and 18-21



Further documents are listed in the continuation of Box C.



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A document defining the general state of the art which is not considered to be of particular relevance	*X* document of particular relevance; the claimed invention cannot be considered novel or cannot be considered to involve an inventive step when the document is taken alone
B earlier document published on or after the international filing date	*Y* document of particular relevance; the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art
L document which may throw doubts on priority claim(s) or which is cited to establish the publication date of another citation or other special reason (as specified)	*A* document member of the same patent family
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INTERNATIONAL SEARCH REPORT

International application No.

PCT/US99/16616

C (Continuation). DOCUMENTS CONSIDERED TO BE RELEVANT

[illegible]

PCT

NOTIFICATION OF ELECTION

(PCT Rule 61.2)

From the INTERNATIONAL BUREAU

To:

Assistant Commissioner for Patents
United States Patent and Trademark
Office
Box PCT
Washington, D.C.20231
ETATS-UNIS D'AMERIQUE

in its capacity as elected Office

Date of mailing (day/month/year)

13 April 2000 (13.04.00)

International application No.

PCT/US99/16616

Applicant's or agent's file reference

Hawtof 7-2

International filing date (day/month/year)

22 July 1999 (22.07.99)

Priority date (day/month/year)

07 August 1998 (07.08.98)

Applicant

HAWTOF, Daniel, W. et al

1. The designated Office is hereby notified of its election made:



in the demand filed with the International Preliminary Examining Authority on:

04 February 2000 (04.02.00)



in a notice effecting later election filed with the International Bureau on:

2. The election ☒ was



was not

made before the expiration of 19 months from the priority date or, where Rule 32 applies, within the time limit under Rule 32.2(b).

The International Bureau of WIPO
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